6P01/ Byrm Butler

Analysis of Hard Thin Film Coating

7N-27-CR
118000

Final Report

For NASA-UAH contract NAS8-38609, D.O. 59

Funding level: $19,000 (total)

PI: Dr. Dashen Shen
University of Alabama in Huntsville
Huntsville, AL 35899
(205) 890-6136
FAX: (205) 890-6803
e-mail: shen@ebs330.eb.uah.edu

1. Introduction

MSFC is interested in developing hard thin film coating for bearings. The wearing of the bearing is an important problem for space flight engine. Hard thin film coating can drastically improve the surface of the bearing and improve the wear-endurance of the bearing. However, many fundamental problems in surface physics, plasma deposition, etc, need further research. The approach is using electron cyclotron resonance chemical vapor deposition (ECRCVD) to deposit hard thin film on stainless steel bearing. The thin films in consideration include SiC, SiN and other materials. An ECRCVD deposition system is being assembled at MSFC.

2. Objective

The objective of this work is to provide an initial analysis on the thin film material properties versus the deposition conditions for two material systems: SiC and SiN. The result will be used to determine the direction of further research.

3. Scope of Work

The film is planned to be deposited at MSFC. The measurement should be performed by UAH personnel using MFSC equipment. The analysis should be carried out at UAH. Since the deposition and measurement is an interactive procedure, (the information from the data analysis is used as feedback), we expect frequent on-site visits.

4. Schedule

The original program covers a one year period (Jan. 1992 - Dec. 1992). However, due to the delay in system assembling caused by the budget difficulty at MFSC, the program has been extended several times (no-cost extension). In fact, the system is still being assembled now.

5. Results

According to the situation, our major effort is switched to following two tasks:

1. Provide assistance on ECRCVD system assembling at MSFC

2. Literature search: collecting and studying the information on the hard film coating.

Dr. Shen stayed for a period at MSFC in summer, 1992, to assist system assembling. He also visited MSFC and discussed the project with MSFC personnel numerous times. Currently, the system already shows a good vacuum in pump-down, but need more work to start deposition.

Information on the application of hard thin film coating is collected. We believe the approach is promising. Section 6 is a proposal for further investigation. Important applications are summarized in the proposal. We sincerely hope MSFC can fund this work, but we are also interested in submitting the proposal with MAFC to other funding agencies.

6. Proposal for further investigation

Summary

Corrosion and wear of mechanical and structural components and systems are an important issue for NASA, US armed forces and industry. Surface coatings are often used to protect these components. However, the coating process often leads to the generation of hazardous wastes.

Silicon based thin coatings are used in the semiconductor and flat panel display industries for various applications. Many of these films have refractory properties but can be applied at low temperatures. These films have the potential of being developed into corrosion and wear resistant coatings. Techniques for their deposition over large surface areas have been developed. Using these silicon-based thin coatings can improve performance, decrease costs, and cause only minimal environmental impact.

We propose to concentrate on the application of thin, transparent silicon based coatings on plastics and metals. The films under consideration include: SiO_2, SiN, SiON, SiC, BN and their combinations in the form of multi-layer composites

We will use ECRCVD as our deposition method. ECRCVD has a number of special advantages: capability of deposition of refractory films on plastic substrates; strong bonding to substrate by substrate pre-cleaning in a glow discharge and by ion bombardment during deposition; formation of metal silicide bonding layers on appropriate substrates; demonstrated large area and curved-surface capability.

The program will include three major tasks:

1. Optimize processing conditions: we will optimize processing conditions, including deposition temperature and gas dilution. We will also develop in-situ surface cleaning and surface treatment technology for this application.

2. Research on the corrosion and wear resistant properties of various films: we will deposit silicon based films, including SiO_2, SiN, SiON, SiC, BN, and evaluate their erosion/corrosion resistance.

3. Thin film multi-layers: we will develop a combined corrosion/erosion and wear resistant coating using multi-layer of silicon based films.

The program covers a four year period. In the first year layers of thin films including SiO_2, SiN, SiON, and SiC will be deposited, and their erosion/corrosion resistance properties will be measured. In the second year the deposition conditions will be optimized according to the erosion/corrosion resistance properties of the films. New films such as BN will be deposited. New liquid source deposition methods will be studied. In the third year thin film multi-layers will be deposited and the optimum layer structures will be studied. In the last year a demonstration will be given.

The research team will include MSFC personnel, Prof. Shen at The University of Alabama in Huntsville, and graduate students.

Technical Approach

We will develop proper technology for using silicon based coatings for erosion/corrosion and wear resistant coating. The films under consideration include:

- SiO_2 (silicon dioxide)
- SiN (silicon nitride)
- SiON (silicon oxynitride)
- SiC (silicon carbide)

We will use ECRCVD as our deposition method. Because ECRCVD can be extended easily to the deposition of boron nitrides, this coating will also be considered. ECRCVD has a number of special advantages:

- Capability of deposition of refractory films on plastic substrates

- Strong bonding to the substrate by substrate pre-cleaning in a glow discharge and/or by ion bombardment during deposition

- Formation of metal silicide bonding layers on appropriate substrates

The program will include three major tasks:

1. Proper processing conditions

Currently, many processing conditions in ECRCVD are optimized for applications in semiconductors or thin film electronics. To adapt these thin films to erosion/corrosion resistant coatings, the proper processing conditions will be defined.

1.1 Deposition temperature

For coatings on plastic or polymer materials, a low processing temperature is important. The typical ECRCVD temperature for electronic application lies between 200°C and 300°C. When the substrate temperature is lower, the surface mobility of the pre-cursors is low, thus the film tends to have microstructures on the 10 nm scale.

According to our experience, the film quality can be drastically improved by using heavy dilution, with hydrogen or other carrier gases (He or Ar). In heavy dilution, gas molecules cover the growth surface, and an improved surface mobility results. In this program we will develop this dilution technology to adapt it to corrosion and wear resistant coatings for plastic and polymer components.

Higher processing temperatures can be used for the coatings of metal components. We will seek proper processing conditions which can not only provide corrosion resistant coatings, but also improve performance, such as wear resistance. We also will pay special attention to the direct formation of protective metal silicides during the deposition process.

1.2. In-situ surface cleaning

In-situ surface cleaning technology will be developed. In-situ plasma etching or sputtering are used in the semiconductor and flat panel display industries to clean the substrate surface to ensure strong bounding of coatings. We will develop proper processing conditions for in-situ cleaning methods with hydrogen, helium or argon discharges, in preparation for coating. We also will develop low-energy ion bombardment as a pre-cleaning tool. We plan to mount the substrate on the powered electrode, and using a bias voltage, ion bombard the substrate to improve cleaning efficiency.

1.3. Surface treatment

Ion implantation can be used to modify the surface of materials. However, an ion implanter is expensive and not suitable for large area coating. Recently, the ion-shower was developed by the flat panel display industry for large area doping. The method uses an ion shower and electrical bias, but no mass separation. Therefore, it could be used as a low cost surface treatment for coating applications. We plan to explore this approach.

2. Erosion/corrosion and wear resistant properties of various silicon based films

Several silicon based thin films, including SiO_2, SiN, SiON and SiC can be used for erosion/corrosion resistant coatings. We will deposit these films, plus BN, and optimize and compare their properties.

2.1. Silicon dioxide (SiO_2)

ECRCVD SiO_2 can be deposited at room temperature, using a gas mixture of SiH_4, and N_2O diluted in He. We were involved in a project where such SiO_2 was deposited on polymer parts for the car industry. In the proposed program we would like to explore using SiO_2 as erosion/corrosion resistant coating. Deposition conditions will be optimized, and the erosion/corrosion resistance will be measured.

2.2. Silicon nitride (SiN)

ECRCVD SiN is an excellent refractory and diffusion barrier. Deposited under proper conditions, the film also is resistant against acid-etching. SiN can be deposited from a gas mixture of SiH_4, NH_3, N_2 and H_2. The Si/N ratio and therefore the chemical property of the film can be adjusted through varying the gas mixture ratio. In this program we will develop the proper deposition conditions for erosion/corrosion resistance, and characterize it.

2.3. Silicon oxynitride (SiON)

One advantage of ECRCVD deposition is that the gas mixture can be very flexible, thus the content of the film can be easily adjusted. For example, using a mixture of SiH_4, N_2O, CO_2 and NH_3, SiON can be deposited. The erosion/corrosion resistant of such films is not known. It is were measured by FTIR and compared with the intensity of H_2O bonds before soaking. Since the method can provide quick feedback, it is very useful for optimizing film deposition conditions.

The coatings must be tested under realistic conditions. A weather chamber to simulate these conditions will be set up.

Advanced analytical tools such as scanning electron microscopy will be use to study the surface and interface.

3. Multi-layer thin films for combined erosion/corrosion and wear resistance

Another advantage of ECRCVD is that it is easy to make multi-layer coatings. Switching the composition of the feedstock gas produces layers with different chemical composition and mechanical properties. Graded layers also can be fabricated by appropriate mass flow control. In this program we will build composite coatings for combined erosion/corrosion and wear resistance. For example, SiC layer can be deposited to provide wear protection, on top of SiN layer which provides a vapor barrier.

It is important to point out that in principle the technology already exists for fully automated control of deposition. By what is called inverse modeling in integrated circuit technology, the user can input the erosion/corrosion resistance requirement, and then software can design a proper layer combination and thickness. The ECRCVD mass flow controllers can be controlled by computer, so that the layers can be deposited automatically.

A detailed plan will be submitted if a formal proposal is invited.